



PATENT
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IN THE U.S. PATENT AND TRADEMARK OFFICE

In re application of

Nevio FRANCESCUTTI et al.

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PROCESS FOR MONONITRATION OF ALKANEDIOLS

DECLARATION UNDER 37 C.F.R. §1.132

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Sir:

I, Nevio FRANCESCUTTI, a named inventor, am a citizen of Italy and reside at Via Caravaggio, 21, San Giovanni Di Casarsa, Italy.

I am familiar with the above-identified U.S. patent application including the Examiner's position that claims 1-9 of the application are obvious over GB 1,040,139 and that claim 10 of the application is obvious over GB 1,040,139 in view of U.S. Patent 1,596,622 and U.S. Patent 2,294,849.

In order to demonstrate the non-obviousness of the claims of the application, the following experimental measurements have been performed:

EXAMPLE A

Example 3 of GB 1,040,139 was repeated using butanediol (BD), instead of pentaerythritol, with the highest molar ratio of nitrating agent/substrate taught by GB 1,040,139, i.e., 7.55:1, instead of 6.70:1. After 10 minutes from the addition of sulfuric acid, the reaction mixture decomposed with the production of nitrous vapors.

EXAMPLE B

Example 1 of GB 1,040,139 was repeated using BD instead of pentaerythritol, in the same molar ratio, i.e., 7.55:1, and with nitric acid as the nitrating agent. After 18 minutes, the reaction was quenched in water. Butanediol-mononitrate (BDMN) was obtained in a molar yield of about 14.7%, and butanediol-dinitrate (BDDN) was obtained in a molar yield of about 20.3% as a byproduct.

EXAMPLE C

Example B was repeated, and after 1 hour the reaction was quenched in water. BDMN was obtained in a molar yield of about 17.4%, and BDDN was obtained in a molar yield of 16.5% as a byproduct.

EXAMPLE D

Example C was repeated and the reaction was quenched in water after 2 hours. BDMN was obtained in a molar yield of about 19.9%, and BDDN was obtained in a molar yield of about 15.5% as a byproduct.

Conclusion

These results demonstrate that that the nitration of butanediol according to GB 1,040,139 will fail to achieve mononitration, because the reaction yields a considerable amount of dinitration product. In particular, these experiments demonstrate that:

- a mixture of nitric and sulfuric acid is not suitable for the mononitration of alkanediols, and it should be noted that even though GB 1,040,139 teaches that mononitration can be carried out using nitric acid alone or in admixture with sulfuric acid, all the examples entail the utilization of sulfuric acid;

- GB 1,040,139 teaches that the optimal period for the completion of the reaction is between 1 and 2 hours, while the process of the present invention is carried out for a time ranging from 10 to 30 minutes; and

- GB 1,040,139 teaches that the molar ratio between the nitrating agent and the substrate should range from 4.88:1 to 7.55:1, but Examples B and C demonstrate that

this ratio will yield a considerable amount of dinitration product.

Accordingly, the effective mononitration of alkanediols is surprising and represents an unexpected result over GB 1,040,139. The present invention results from the inventive selection of several specific reaction conditions that lead to the mononitration of alkanediols of formula (II), namely:

- the lack of sulfuric acid in the nitrating agent;
- the concentration of nitrating nitric acid;
- the utilization of nitric acid substantially free from nitrous acid and nitrogen oxides;
- the weight ratio of nitric acid to the compound of formula (II); and
- the contact time between nitric acid and the compound of formula (II).

Therefore, any obviousness that may be alleged over GB 1,040,139 is fully rebutted by the unexpected results of the present invention.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are

punishable by fine or imprisonment, or both, under \$1001 of Title 18 of the United States code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date _____

Nevio FRANCESCUTTI